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A Preliminary Evaluation of the Phosphazene Resin PPZTM

Adam J. Jaglowski, Karim M. Gabriel,
and Marc S. Pepi

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| 13. ABSTRACT (Maximum 200 words) A phosphazene material, PPZ™, is reported by Idemitsu Petrochemical Company (Tokyo, Japan) to have excellent chemical and abrasion resistance properties for hardcoating applications. An evaluation of the chemical resistance of this coating is presented. PPZ coated on poly(ethylene terephthalate) (PET) films were challenged with simulants (2-chloroethyl ethyl sulfide (CEES), 2-chloroethyl methyl sulfide (CEMS), diisopropyl methylphosphonate (DIMP)) and a decontaminant (DS-2) via a droplet test method. Preliminary results showed that the PPZ coating was affected by DIMP, CEES, and CEMS as observed by optical and scanning electron microscopy (SEM). Upon visual examination, the DS-2 treated specimen did show an effect; however, the coating's behavior to this corrosive liquid is inconclusive. In addition, PPZ™ resin was coated on polycarbonate (PC) using a modified ASTM D823-87 method and cured by ultraviolet light. Curing time was determined by Fourier transform infrared spectroscopy (FTIR) using an attenuated total reflectance (ATR) technique. The Taber abrasion resistance of the PPZ-coated PC specimens were slightly lower than reported values. | | | | |
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Introduction

Idemitsu Chemical Company has developed a curable phosphazene resin (PPZ™) (1-4), which is being marketed as a hardcoating for various substrates such as poly(ethylene terephthalate) (PET), poly(vinyl chloride) (PVC), poly(methylmethacrylate) (PMMA), polycarbonate (PC), and wood. The cured resin is reported to have the following properties:

- Excellent Transparency
- Superior Chemical Resistance
- Substantial Abrasion Resistance
- Extreme Hardness
- Excellent Heat Resistance
- Low Curing Shrinkage
- Low Dynamic Friction Coefficient

The cured phosphazene resin is observed to be harder and more abrasion resistant than PMMA and PC. The phosphazene resin is also reported to be chemically resistant to detergents, bleach, solvents, dilute acid, and alkaline solutions (1,2).

PC is widely used in the Army for transparent armor applications such as lenses and canopies because of its optical clarity and ballistic properties. However, better abrasion and chemical resistance properties of PC are needed. Work has been undertaken to evaluate potential protective hardcoatings for PC (5). Since the PPZ material appears to have good mechanical, chemical, and optical properties and can be applied to PC substrates, an evaluation is warranted. This report describes the chemical resistance evaluation of the phosphazene resin PPZ (on PET substrate) to DS-2, 2-chloroethyl ethyl sulfide (CEES), 2-chloroethyl methyl sulfide (CEMS), and diisopropyl methylphosphonate (DIMP). In addition, this report also describes the coating of PC with PPZ and abrasion testing of these PPZ-coated specimens.

The resin is composed of the material, 3PNC-6HEMA 1 which is a cyclic phosphazene. 3PNC-6HEMA is prepared from the reaction of hexachlorocyclotriphosphazene with 2-hydroxyethyl methacrylate (see Figure 1). The phosphazene monomer can be cured by irradiation with ultraviolet (UV) light, electron beams, or heat.

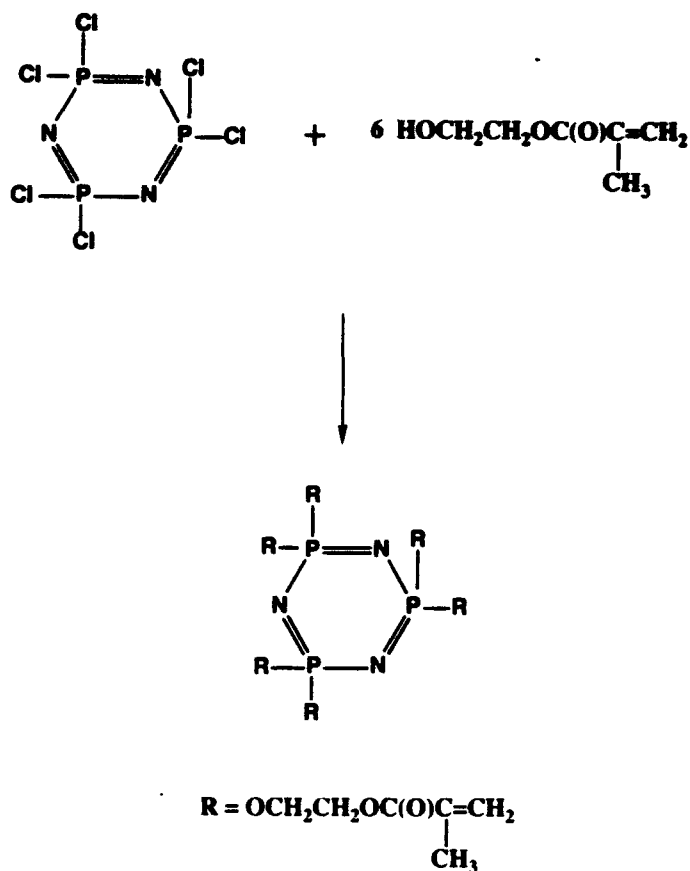


Figure 1. Preparation of 3PNC-6HEMA 1.

Experimental

Materials

Uncured phosphazene resin (PPZTM resin, Lot No. 911211-U-1000) and PPZ coated on 120 μm PET films (8 in. x 11 in.) were obtained from Idemitsu Petrochemical Company, Japan (POC: Dr. Atsunori Yaguchi). The thickness of the PPZ coating on PET was approximately 10 μm . CEES, CEMS, and DIMP were used as-received. DS-2 was prepared by mixing 2% sodium hydroxide, 28% 2-methoxyethanol, and 70% diethylenetriamine by weight. The DS-2 mixture was used immediately after preparation. Polycarbonate sheets (G. E. Lexan) coated with a protective paper on both sides were cut to 4 in. x 4 in. x 0.125 in. dimensions for coating.

Instrumentation

A Fourier transformed infrared (FTIR) spectrum of uncured PPZ resin was recorded in percent transmittance with a Perkin-Elmer 1740 Infrared Fourier Transform Spectrophotometer at 4 cm^{-1} resolution as a thin film between NaCl plates. FTIR spectra of PPZ-coated PET and PC samples were obtained with the same spectrometer and an Analect Instrument ATR Model FXA-523 4265 accessory, equipped with a 45° KRS-5 crystal (thallium bromide iodide), at an incident angle of 45°.

Gas Chromatography/Mass Spectrometry (GC/MS) was performed on a concentrated solution of PPZ resin in acetone with a Hewlett-Packard 5995C GC/MS using helium as the carrier gas, a split ratio of 50:1, and a 0.33 μm poly(dimethylsiloxane) stationary phase. The fused-silica capillary GC column (12 m, 0.20 mm) was temperature programmed from 30°C to 320°C at 10°C per minute. The mass spectrometer was scanned from mass/charge (m/z) 10 to m/z 400 at a rate of 2 scans per second.

Optical micrographs were obtained with an Olympus SZH stereomicroscope. Scanning electron micrographs (SEM) were obtained with a JEOL JSM-840A SEM using an accelerating voltage of 15.0 KeV. SEM samples were sputtered with a gold/palladium coating before examination.

Chemical Droplet Testing

Single droplet testing

In a vented hood, a 2 in. \times 2 in. sample of PPZ-coated PET was challenged with one of the following liquids: CEMS, CEES, DIMP, or DS-2. A 3 microliter droplet of a challenging liquid was applied to the center of the sample and a watch glass was then placed over the sample. After 24 hours at room temperature, the watch glass was removed, and the sample was left uncovered for an additional 24 hours for aeration. Each sample was thoroughly rinsed with distilled water and allowed to dry before microscopic examination.

Multiple droplet testing

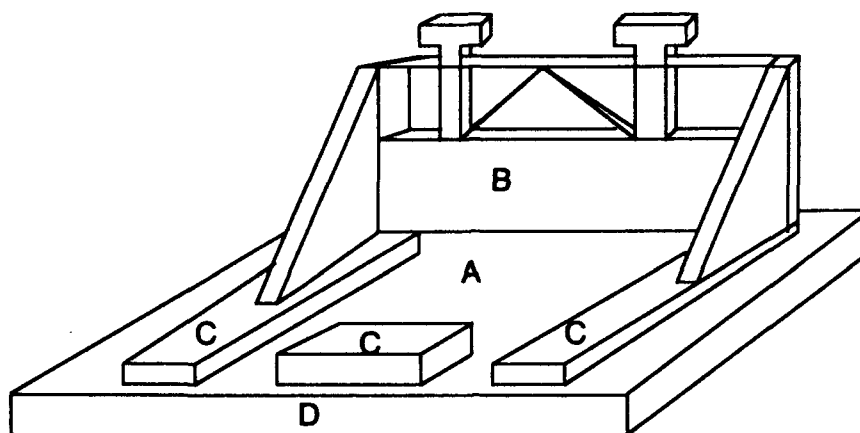
The procedure was the same as the single droplet testing except that a 3 microliter droplet of a challenging liquid was applied four times, once every 24 hours.

Coating of PC Substrate

One side of the protective laminate was removed from the PC prior to coating. The exposed side was washed several times with isopropyl alcohol and air dried with a heat gun. The exposed PC substrate was coated using a modified method of ASTM D 823-87 (6) and a Gardener knife (see Figure 2). The uncoated sample was held firmly in place by three PC sheets of the same thickness which were secured with double-sided tape to an underlying glass surface. The sheets provided a virtually frictionless path along which the knife could be dragged. After the sample was placed in the channel, the knife edge was placed in contact with the edge of the sample. An empirically determined amount of the PPZ resin was then placed at the edge of the sample in front of the knife. The knife was slowly dragged across the surface of the sample. The thickness of the applied coating was approximately 50 μm .

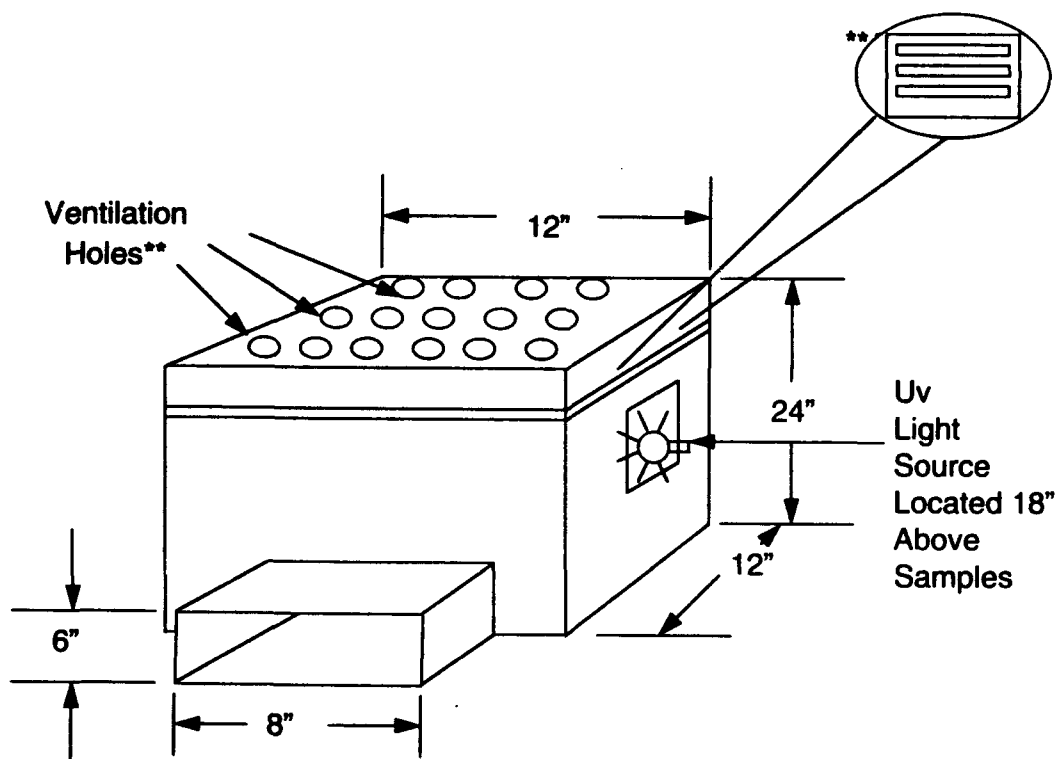
Curing of PPZ-coated PC

The coated PC sample was placed in a UV curing apparatus (see Figure 3) after the underside protective laminate was removed. The UV curing apparatus was constructed of foam board and internally lined with aluminum foil. The sample was then irradiated with a Black-Ray B100-A UV lamp (1600 $\mu\text{W}/\text{cm}^2$) for approximately 15 minutes. The cured PPZ-coated PC samples were cut to specific test sizes and placed between wax paper sheets to prevent scratching and dust accumulation.



A: Sample area
B: Coating knife
C: Polycarbonate guides
D: Glass substrate

Figure 2. Schematic of coating configuration.



*The ventilation holes are offset to allow for free convective cooling of the samples while preventing UV light from escaping.

Figure 3. Schematic of UV curing apparatus.

Taber Test Method and Analysis

Abrasion of coated and uncoated PC samples was performed in accordance with ASTM D 1044-85 utilizing a Taber abrader (7). The samples were cut into 4 in. diameter disks and abraded for 100 cycles with a 500 g load. The samples were then analyzed according to ASTM D 1003-61 (8). The increase in haze was determined with a Gardner integrating sphere haze meter equipped with a PG-5500 Digital Photometric Unit and slit width of 0.469 in.

Results and Discussion

Characterization of Uncured PPZ Resin

The uncured PPZTM resin was analyzed by FTIR and GC/MS. The IR spectrum (see Figure 4a) showed absorptions in the following regions: hydroxyl group (3525 and 1060 cm^{-1}); carbon to hydrogen bond group (3099, 2957, 2894, 1457, and 1408 cm^{-1}); carbon to carbon double-bond group (1638 and 1615 cm^{-1}); carbonyl group (1723 cm^{-1}); carbon to oxygen bond group (1269 and 1176 cm^{-1}), and phosphazene bond group (1234 cm^{-1}). All the noted absorptions should be present for structure 1 with the exception of the hydroxyl group. The presence of the hydroxyl group suggested that the resin is comprised of 1 and other components. GC/MS analysis confirmed that the PPZ resin is indeed composed of several additional components; hydroxylated acrylates and methacrylates, UV curing initiator, antioxidant, diluent, and an unidentified component. However, 3PNC-6HEMA 1 could not be detected by GC/MS since the molecular weight is out of the instrument range of detection. The cured PPZ resin is illustrated in Scheme I. Since the resin is comprised mostly of the methacrylated phosphazene 1 it would likely form a highly crosslinked system.

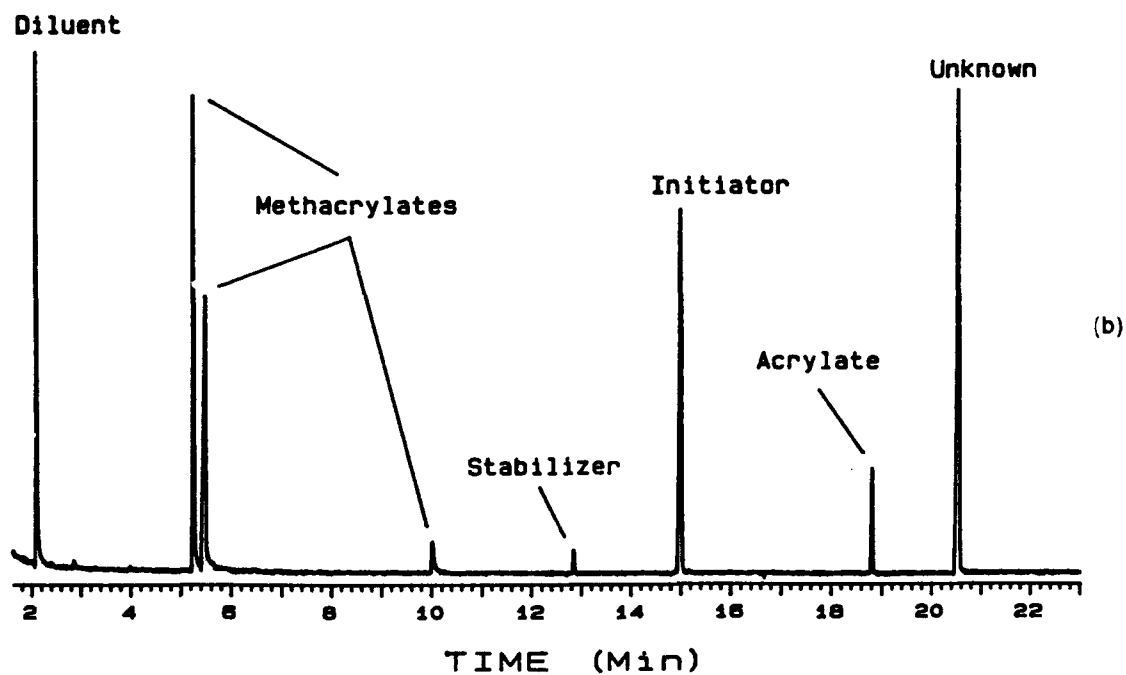
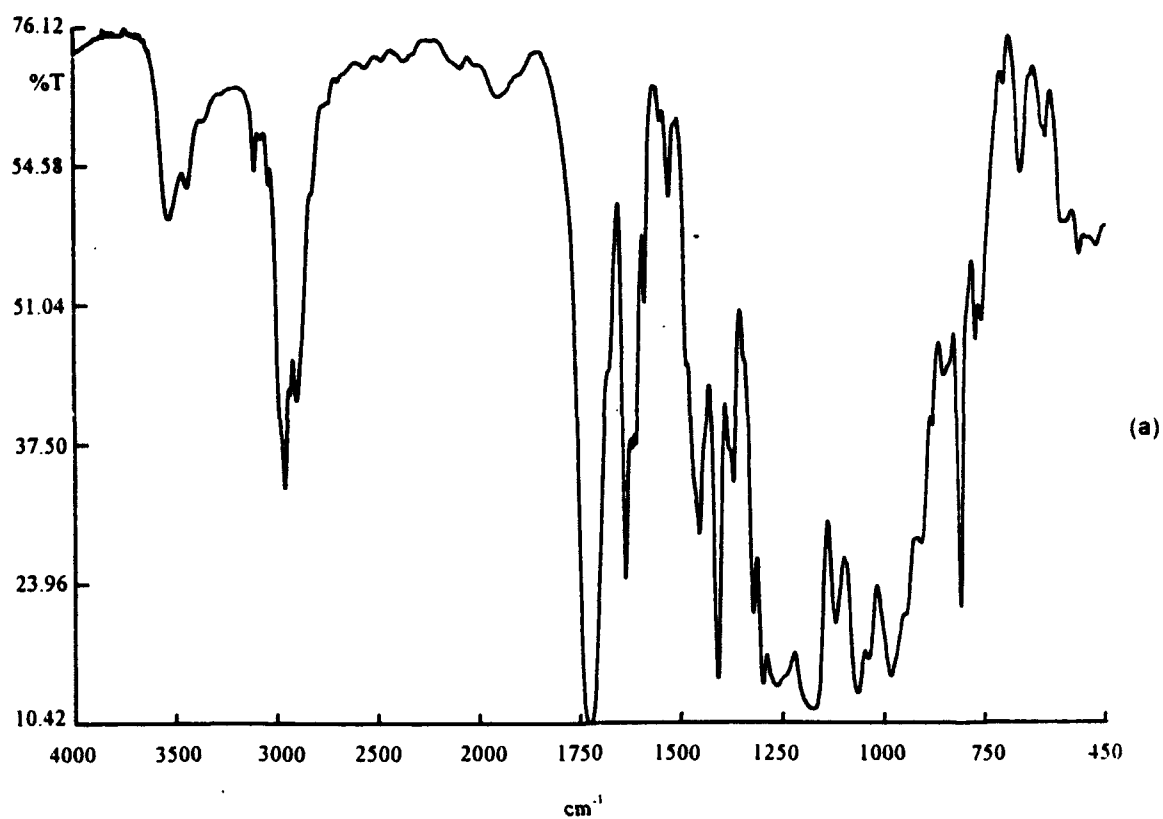
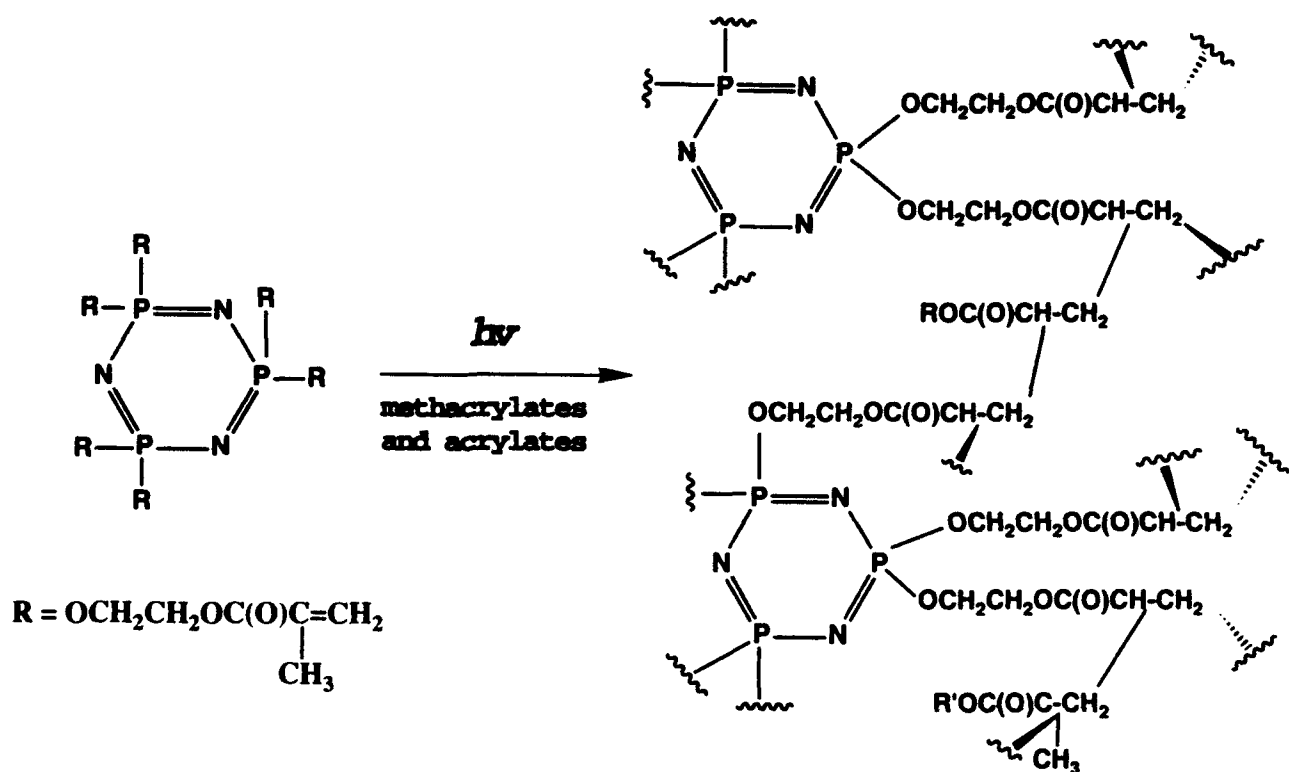


Figure 4. IR spectrum (a) and GC/MS analysis (b) of uncured PPZ resin.

Scheme 1



Chemical Treatment of PPZ-coated PET Films

The PPZ-coated PET samples were exposed to droplets of DS-2, DIMP, CEMS, and CEES. In all instances the challenging liquid affected the PPZ-coated PET samples. The droplets did not spread upon application to the surfaces, with the exception of DIMP. In the DS-2 treatment, only one 3 μL droplet was applied, and a faint hazing was visually observed over the entire area of the applied droplet. An optical micrograph of the effected area was not obtainable. Application of one droplet of DIMP affected the PPZ-coated PET sample in the formation of a ring (see Figure 5a). Upon additional DIMP applications, the liquid migrated to an untreated spot on the film whereby a new ring formed. The PPZ-coated PET samples appeared to be unaffected upon application of one 3 μL droplet of CEMS or CEES; however, multiple droplet additions of CEMS or CEES affected the specimens. The formation of a ring was observed (see Figures 6a and 7a, respectively). How the chemicals affected the PPZ-coated PET films could not be explained by examination of the optical micrographs.

Examination of the untreated PPZ-coated PET by SEM (see Figure 8) indicated that the film had little dimples. These markings may have resulted from the industrial coating process. Whether or not these dimples are sources of pinholes for permeation of liquids to the underlying substrate could not be determined from this study.

A SEM micrograph could not be obtained from the DS-2 treated PET films, and a full explanation cannot be provided to the source of the observed haze in these specimens. Further investigation of the behavior of the coating, by itself, to DS-2 exposure is needed in order to explain this observation. However, it is speculated that the haze was from the underlying PET substrate. Pinholes in the PPZ coating may have allowed the DS-2 to permeate to the PET underlayer. This event is expected to cause an effect on the PPZ coating; however, SEM studies do not reveal any defects on the surface.

The SEM micrographs of the DIMP-treated films showed that upon exposure the affected surfaces developed a cloudlike texture (see Figures 5b through 5d increasing magnification of the same area). This texture gives rise to the observed haze. The treated areas on the PPZ/PET samples did not have the same wettability properties with DIMP as the untreated areas as evidenced by the migration of subsequent droplets to untreated areas. This observation suggested that the surface properties of the treated film changed. There are two possible explanations for this observed change. First, the PPZ coating was modified either morphologically, chemically, or in combination. Secondly, the PPZ coating was removed upon exposure to DIMP and allowed the PET to be exposed. It is known that DIMP does not interact significantly with PET but does with an acrylate (PMMA) resulting in a slow dissolution (9). In a crosslinked acrylate as PPZ, DIMP is not likely to dissolve the PPZ; however, it could swell the coating. If the coating swells and fails, the PET is then exposed. Upon another DIMP treatment, the liquid, which does not interact favorably with exposed PET, migrates to an untreated area on the sample. Application of energy dispersive spectroscopy (EDS) to the cloudlike texture revealed the presence of phosphorous. This result did not support one explanation over the other since, in the latter explanation, it is possible that some PPZ coating material may have been left behind.

In the CEMS- and CEES-treated films, the SEM results revealed that the coating bubbled up in the affected areas, although not completely over the entire treated area (see Figures 6b through 6d and 7b through 7d increasing magnification of the same area). It is well known that acrylates are not chemically resistant to chlorinated hydrocarbons (9,10). Immersion studies of PMMA in a chlorinated hydrocarbon have shown a slow weight loss of the PMMA sample (9). The weight loss is probably due to the dissolution of PMMA in this chemical. Since the PPZ coating has an acrylate functionalized polymer backbone, it was expected that chlorinated hydrocarbons such as CEMS and CEES may interact with the coating, but not to the extent of the uncrosslinked acrylate PMMA. The highly crosslinked PPZ coating was not expected to be soluble in CEES or CEMS. Therefore, the observed bubbling up of the coating was probably due to the adsorption of CEMS or CEES into the coating causing swelling and probably delamination from underlying PET substrate. The observed haze was a result of this bubbling effect.

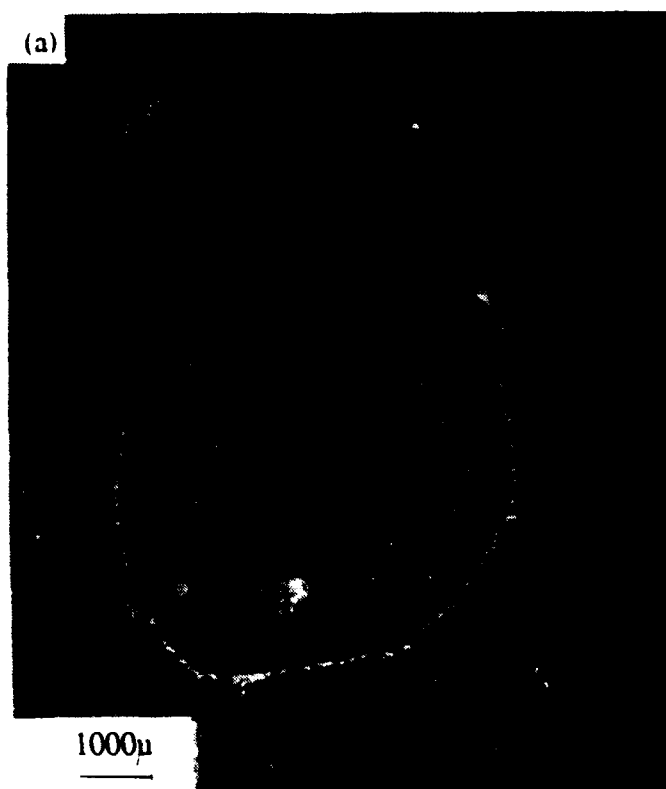


Figure 5. Optical (a) and SEM (b through d) micrographs of DIMP-treated, PPZ-coated PET: (a) 10X; (b) 170X at a 66° tilt angle; (c) 1000X at a 66° tilt angle, and (d) 7500X at a 66° tilt angle

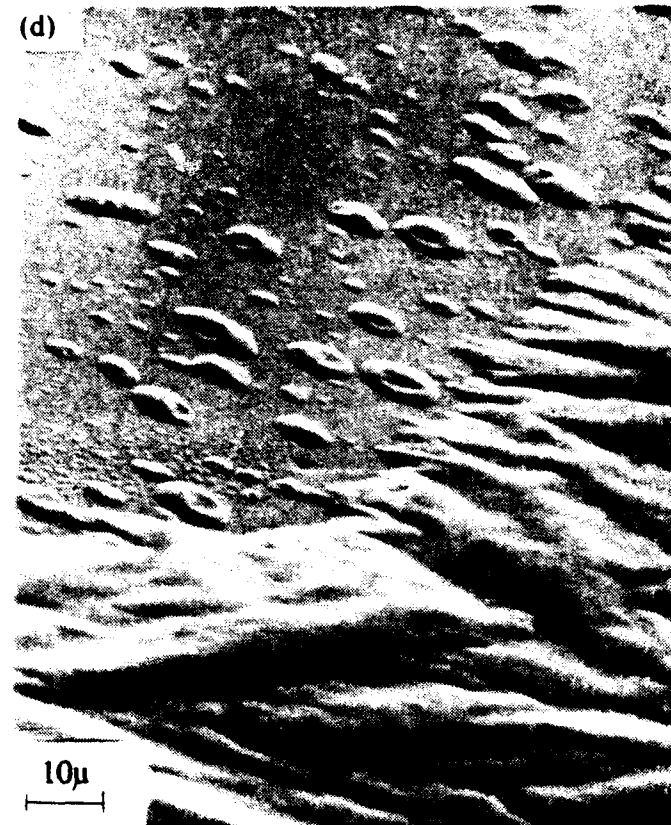
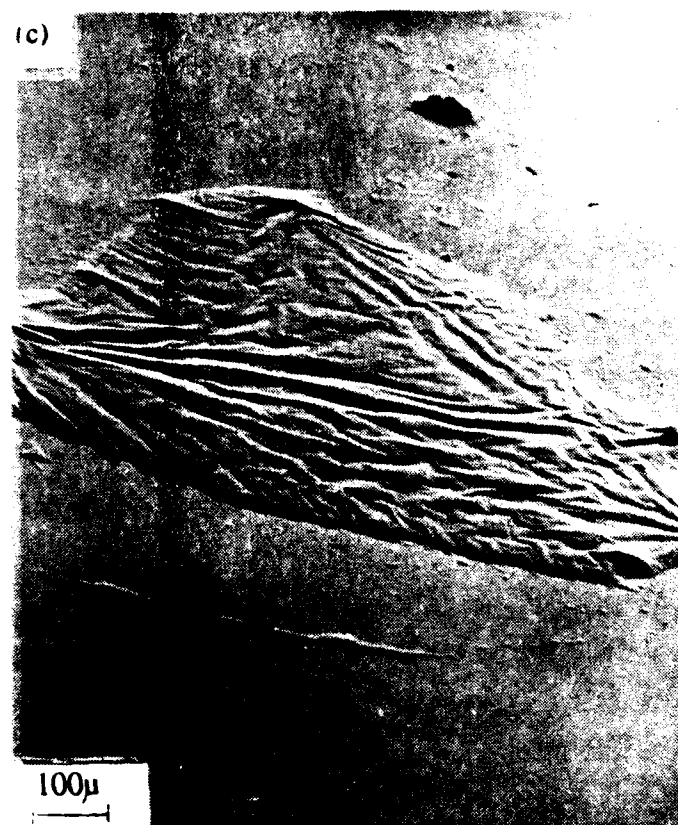
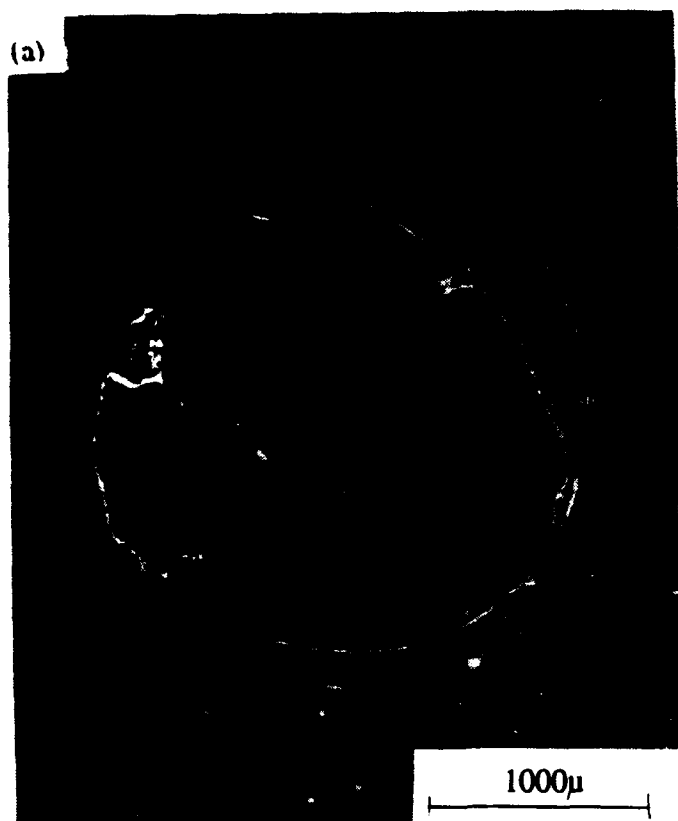


Figure 6. Optical (a) and SEM (b through d) micrographs of CEMS-treated, PPZ-coated PET: (a) 30X; (b) 35X at a 66° tilt angle; (c) 100X at a 66° tilt angle; and (d) 1000X at a 66° tilt angle.

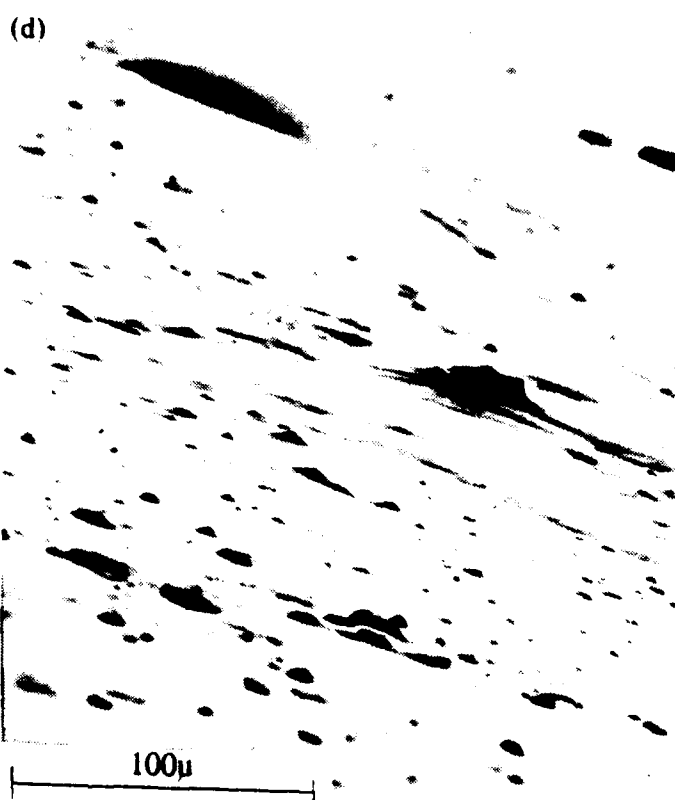
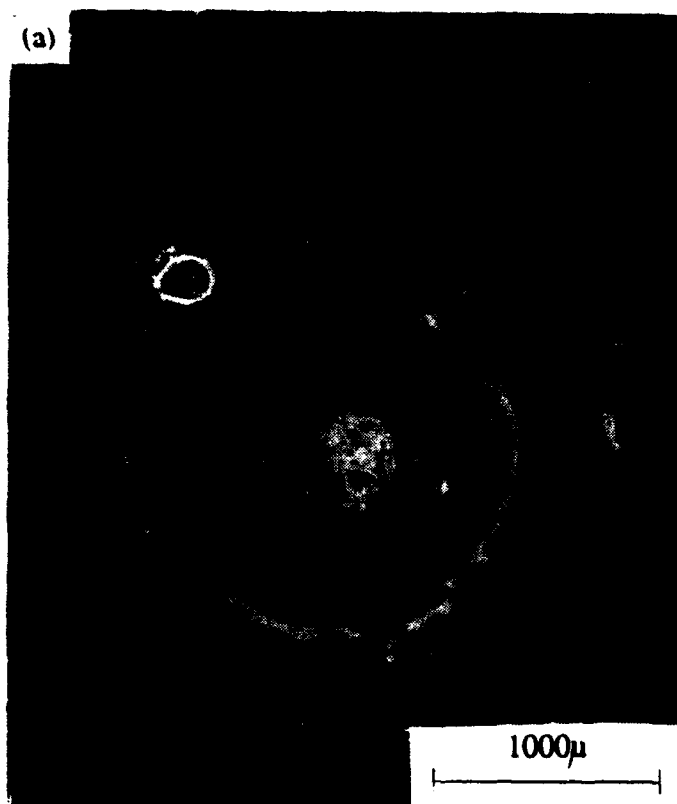


Figure 7. Optical (a) and SEM (b through d) micrographs of CEES-treated, PPZ-coated PET: (a) 30X; (b) 35X at a 66° tilt angle; (c) 170X at a 66° tilt angle; and (d) 400X at a 66° tilt angle.

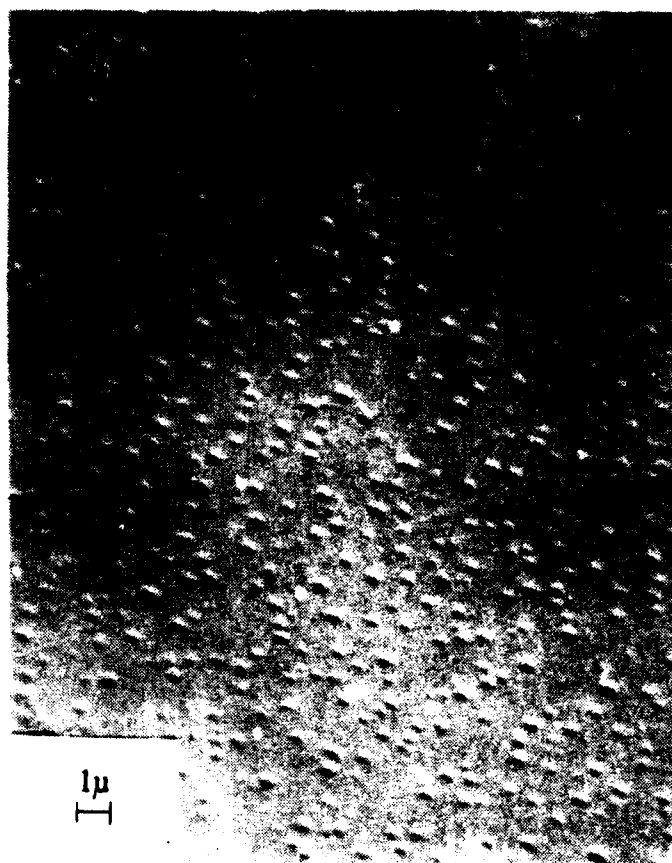


Figure 8. SEM micrograph (5000X) of untreated PPZ-coated PET.

Curing of PPZ Resin on Polycarbonate

The coating and curing of PPZ on PC substrate was accomplished as shown by the schematics in Figures 2 and 3. The curing time for the PPZ resin was determined by comparing the ATR spectra of PPZ coated on PET to specimens cured at times of 10 minutes to 16 minutes (11). The PPZ coating on PET was used as a representative IR spectrum for a fully cured resin (see Figure 9). Figures 10a through 10d show the ATR spectra of cured PPZ resin on PC at UV curing times of 10, 12, 14, and 15 minutes, respectively. In comparing the ATR spectra of the expected fully cured PPZ with cured samples at various times, several things were noted. First, the carbonyl region (1720 cm^{-1} to 1760 cm^{-1}) changed from two medium absorptions to one strong absorption upon an increase in the cure time. Secondly, the 700 cm^{-1} through 1100 cm^{-1} region markedly changed from medium to strong broad absorptions upon an increase in the cure time. Finally, the ATR spectrum of the 15 minute cured specimen resembled the fully cured resin spectrum. Therefore, these results indicated that suitable PPZ-coated PC samples could be prepared using the described coating and curing procedures at a 15 minute cure time. For comparison, the industrial PPZ coating is optimally applied by roll, spray, or dip coating and cured for 3 sec to 10 sec at a higher UV dosage (2).

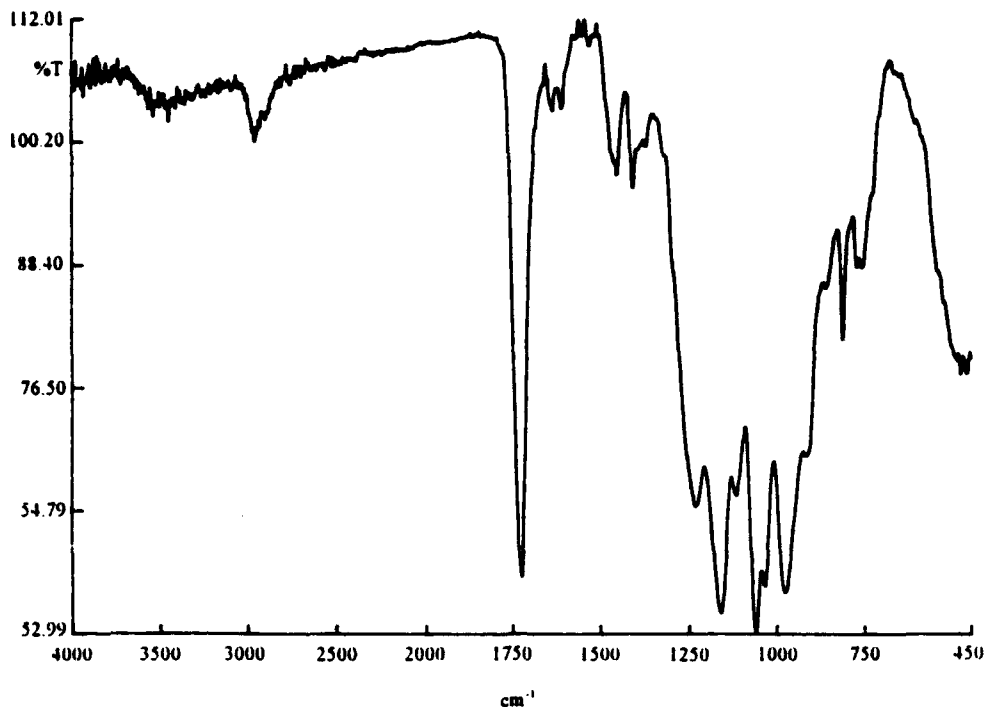


Figure 9. ATR spectrum of the PPZ coating.

Abrasion Testing of PPZ-coated PC

The results of the abrasion resistance tests are summarized in Table 1. These preliminary findings indicated that PPZ did not significantly hinder the transparency of PC. Furthermore, the results show that PPZ provided only a slight increase in abrasion resistance as compared to reported values (2). There are several possibilities for the aforementioned disparity; i.e., curing conditions, coating process, and film thickness. The most apparent cause for the disparity could be improper curing time despite cure monitoring by IR. If the specimens were overcured, then the sample might not perform as desired. An alternative source for the disparity might be the coating process. Lower abrasion resistance could be noted if the samples were poorly coated; this results in nonuniformity, voids, pinholes, or defects. In addition, film thickness could effect the abrasion results.

Table 1. Haze data from Taber abrasion test on polycarbonates

| Sample ID | % Initial haze | % Abraded haze* | % Increase* |
|----------------|----------------|-----------------|-------------|
| PPZ (A) | 2.7 | 28.58 | 25.88 |
| PPZ (B) | 3.9 | 26.83 | 22.93 |
| PPZ (C) | 2.2 | 30.36 | 28.16 |
| PC (A) | 1.2 | 34.05 | 32.85 |
| PC (B) | 1 | 33.9 | 32.9 |
| PC (C) | 0.8 | 30.15 | 29.35 |
| Average of PPZ | 2.93 | — | 25.66 |
| Average of PC | 1 | — | 31.7 |

* These values are the average of multiple readings from the same sample.

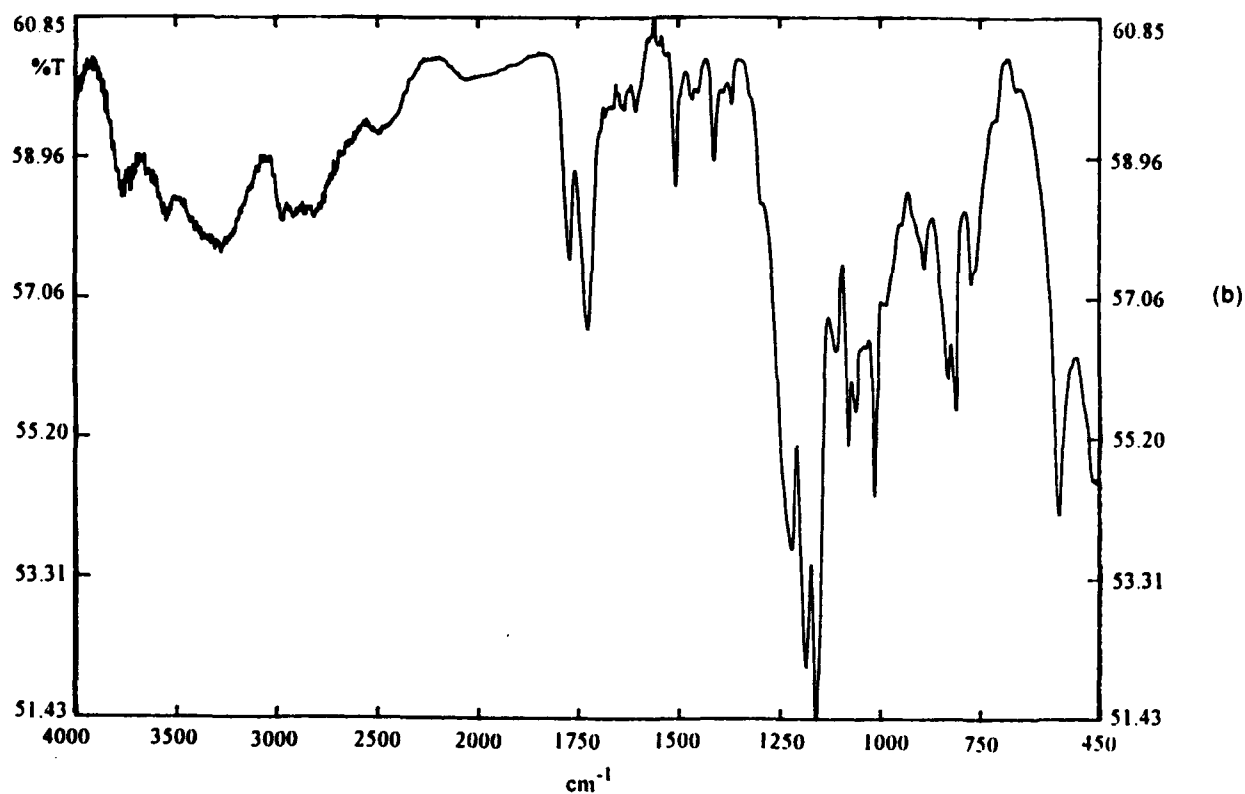
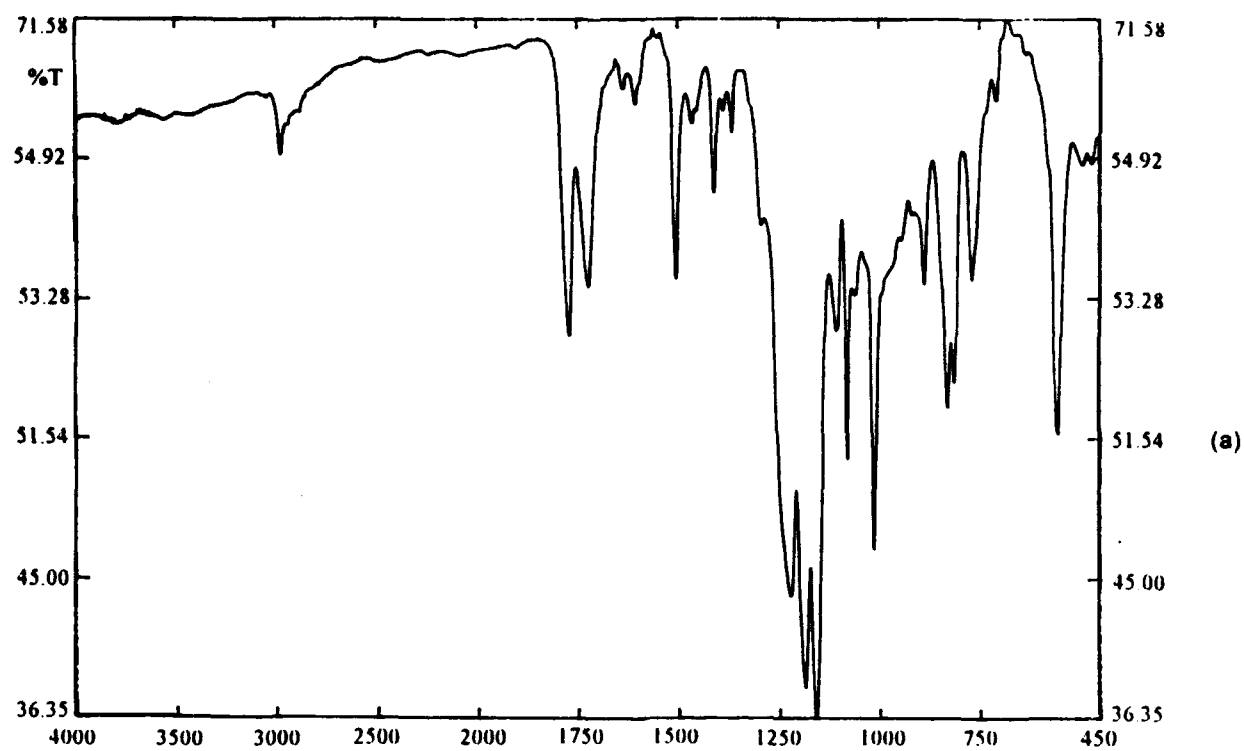


Figure 10. ATR spectra of cured PPZ coatings on PC substrates at curing times of (a) 10 minutes and (b) 12 minutes.

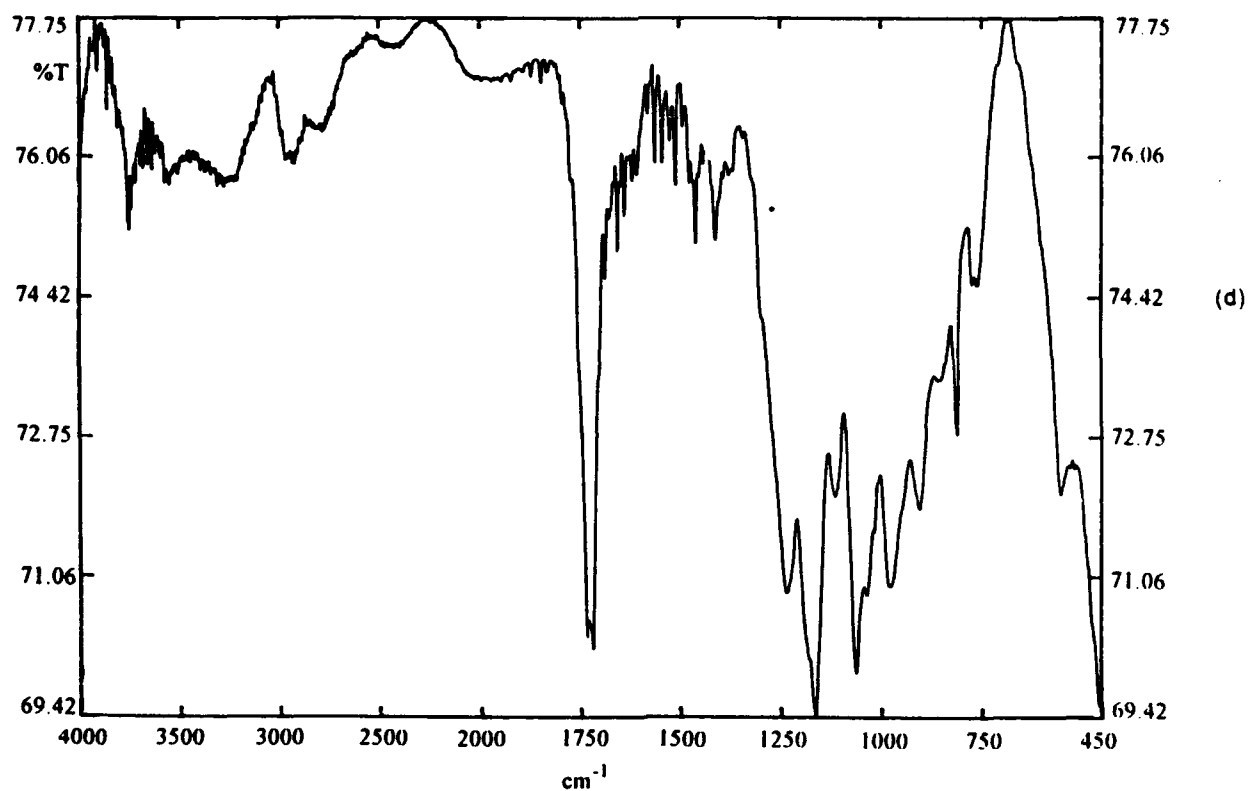
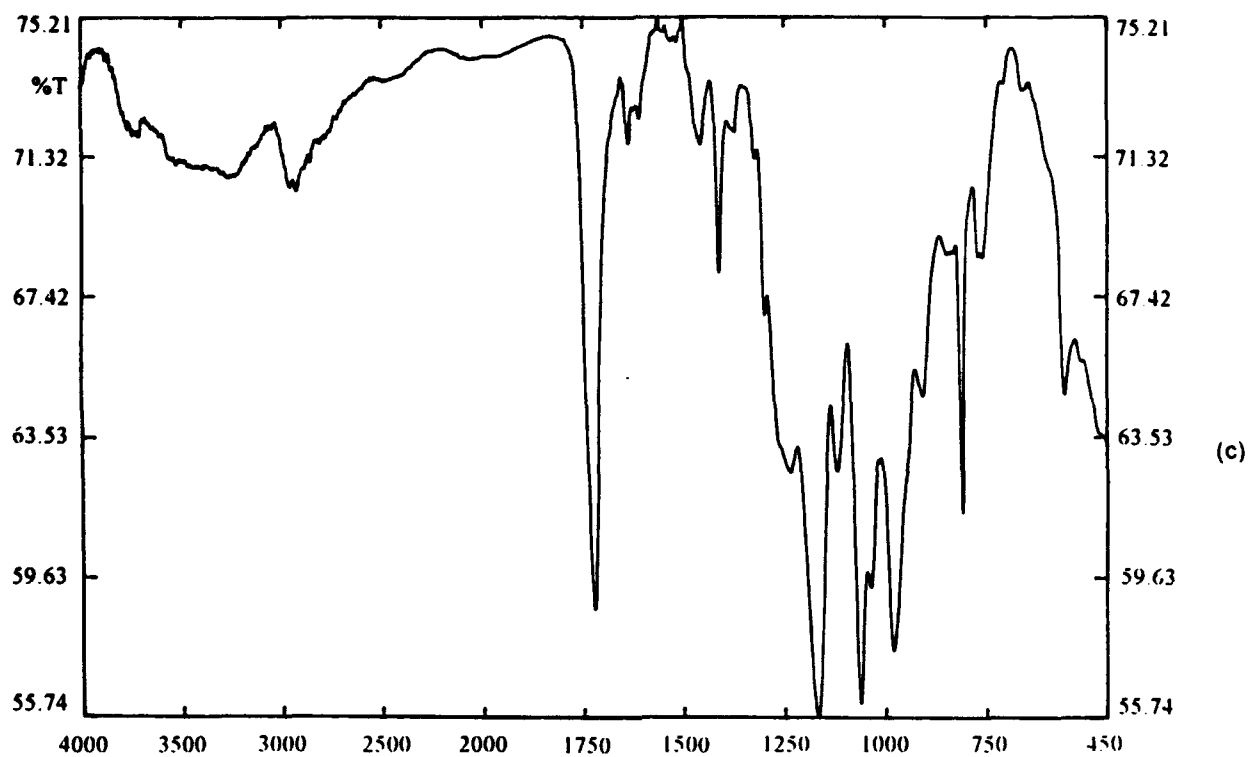


Figure 10. ATR spectra of cured PPZ coatings on PC substrates at curing times of (c) 14 minutes and (d) 15 minutes.

Conclusions

PPZ-coated PET samples were evaluated for chemical resistance to DS-2, CEES, CEES, and DIMP. All specimens were affected; after one droplet addition of DS-2 and DIMP and after multiple droplet additions of CEES and CEMS. This preliminary evaluation revealed that the PPZ coating is effected by CEES, CEMS, and DIMP. In the DS-2 case, additional work is needed to determine if the coating is resistant to this corrosive liquid. Also, further work is needed to fully explain how the DIMP is effecting the PPZ coating. To investigate both of these situations, the PPZ coating must be deposited on a substrate such as glass, which does not interact with the challenging chemical, although these preliminary results suggest that the coating is not chemically resistant to the tested stimulants and decontaminant. Nevertheless, further investigation is needed in order to fully evaluate this coating as a promising material.

PPZ was coated on PC sheets. FTIR monitoring of the curing indicated that for a coating thickness of approximately 50 μm a 15 minute cure was sufficient in the described UV setup. However, evaluation of the abrasion properties with respect to the literature values indicated that the applied coating was not as good as industrially cured PPZ coatings. Further work is needed to fully understand the disparity between our cured samples and the industrially cured coatings.

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